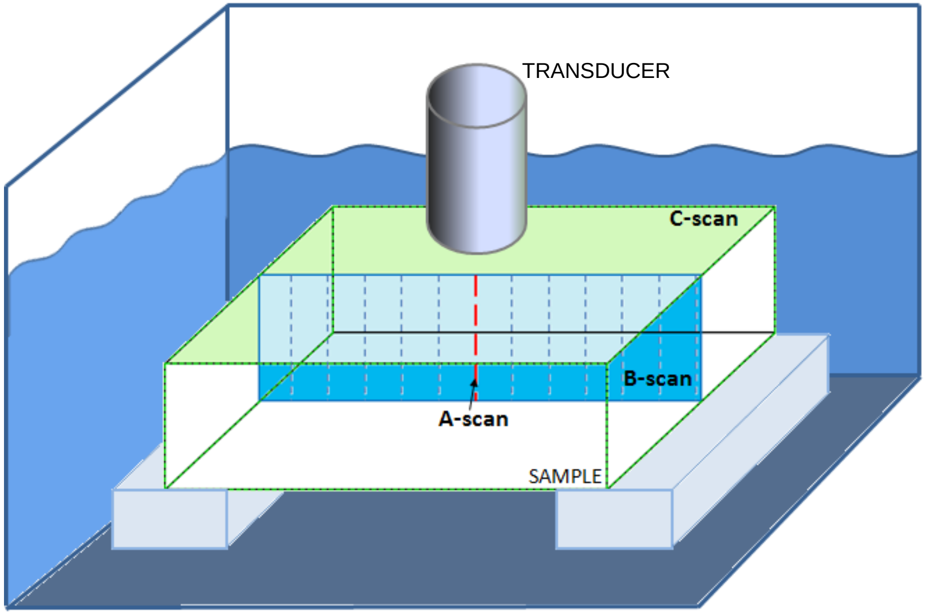
Quantitative Assessment of Homogeneity in SiC Mirrors via Ultrasound NDC

R.A. Haber
V. DeLucca
S. Bottiglieri

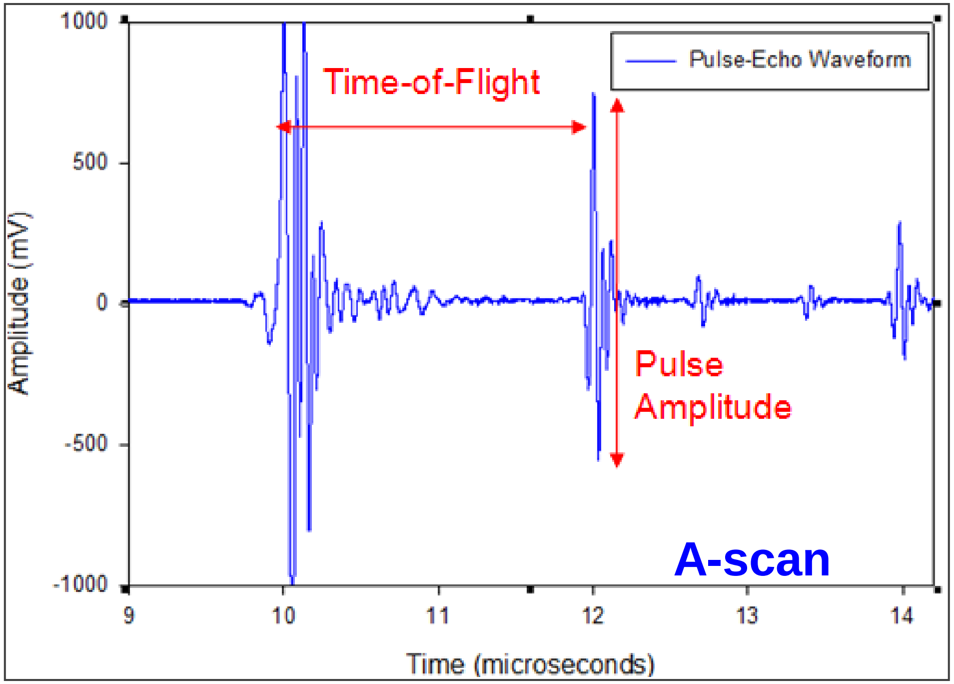
Mirror Days

June 2011

What is Ultrasonic NDE



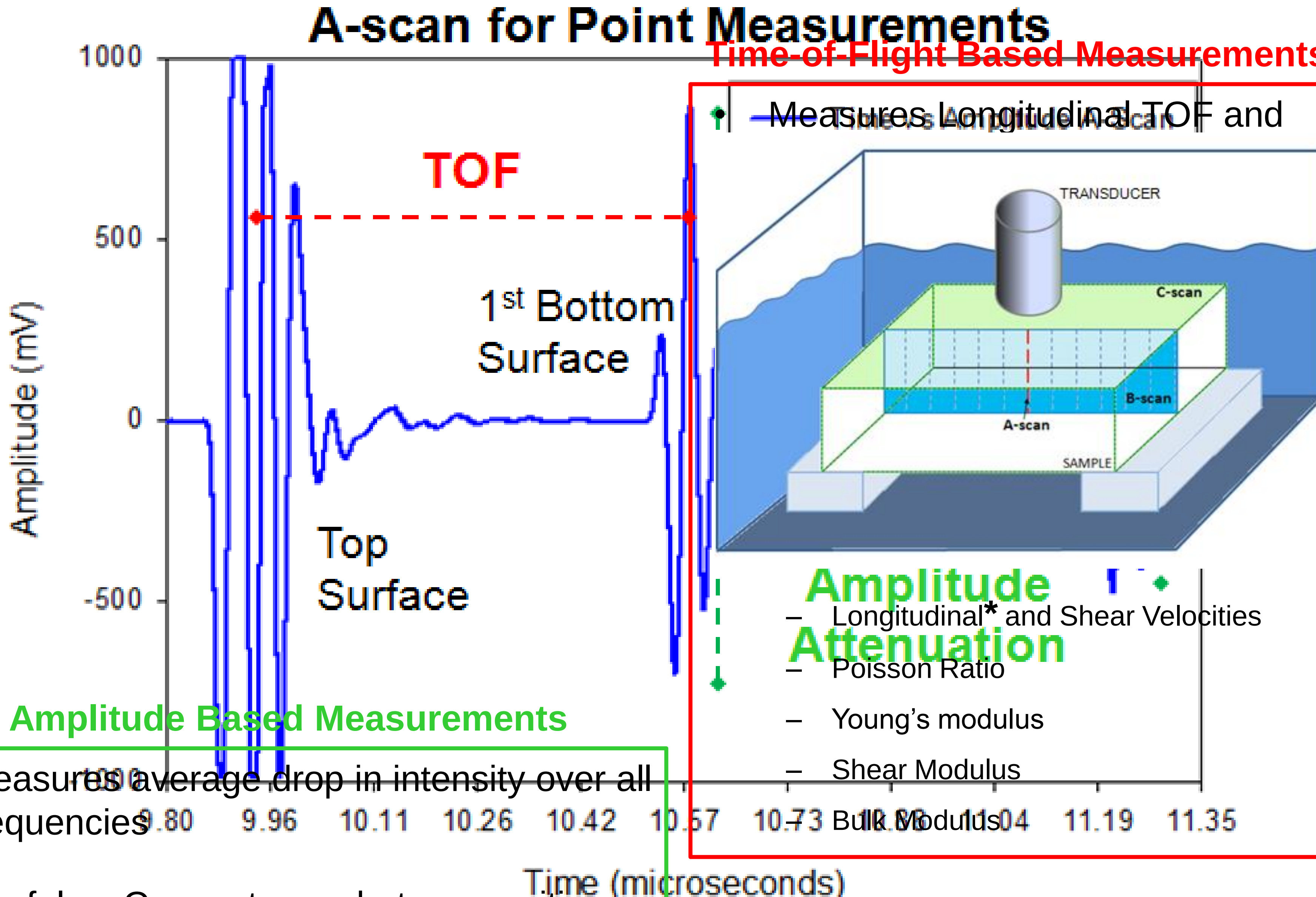
- Immersion-based, pulse-echo system
- C-scan imaging mode useful for mapping material property variations



- Property mapping completed in ~20mins. for a 4” x 4” scan area
- TOF related to c, ρ, ν, E, G, K and Z
- Higher TOF corresponds to lower velocity and therefore lower elastic properties

Amplitude attenuation caused by scattering/ absorption of ultrasound energy

Evaluation-Based Measurements



Time-of-Flight Based Measurements

- Measures Longitudinal TOF and
  - Longitudinal* and Shear Velocities
  - Poisson Ratio
  - Young's modulus
  - Shear Modulus
  - Bulk Modulus

Amplitude Based Measurements

- Measures average drop in intensity over all frequencies
- Useful as C-scan to see heterogeneities

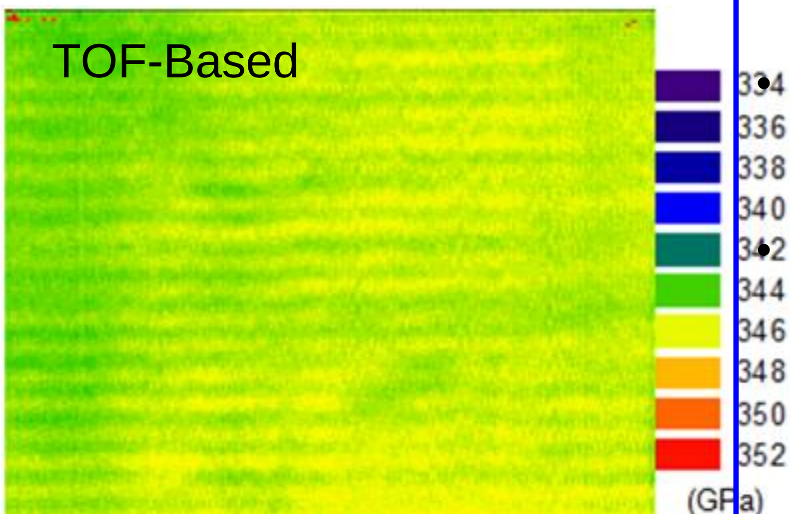
Evaluation-Based Measurements

C-scan Mapping

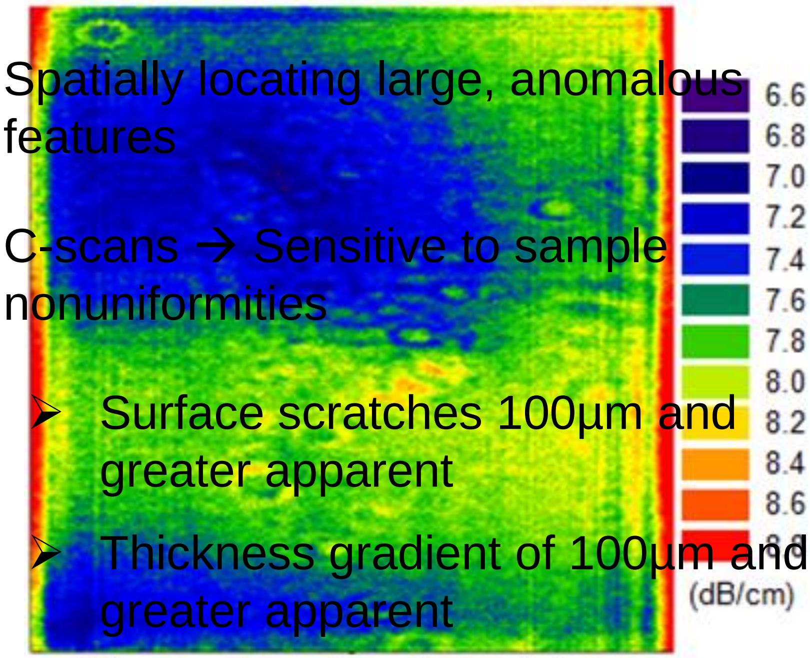
- Determining elastic property variations → Semblance of porosity variations
- Spatially locating large, anomalous features
- C-scans → Sensitive to sample nonuniformities
  - ➢ Surface scratches 100μm and greater apparent
  - ➢ Thickness gradient of 100μm and greater apparent
  - ➢ Sample thickness greater than 2”
  - ➢ Porosity greater than 10%
- 4” x 4” tile scan in 30 minutes

C-scans for Area Property Maps

TOF-Based



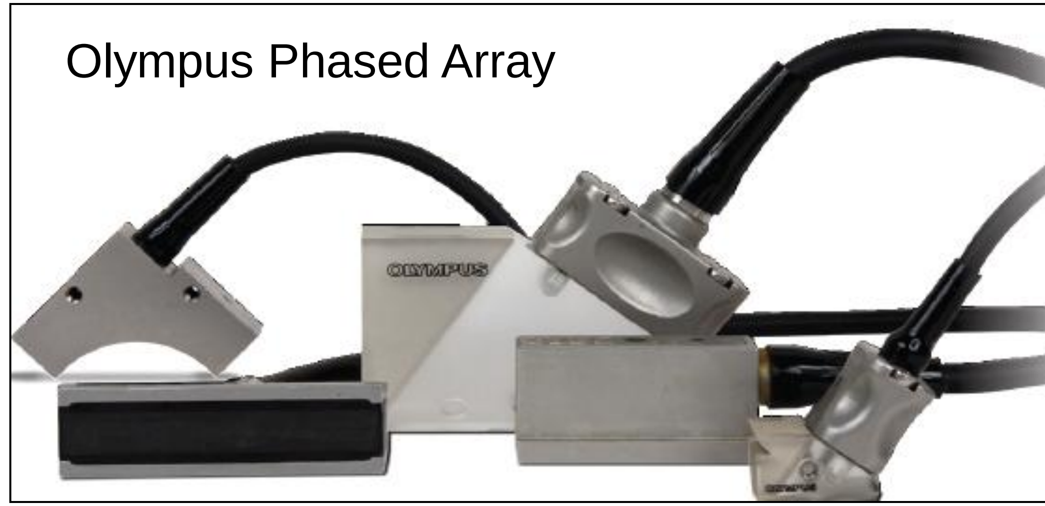
Young’s Modulus Map



Attenuation Coefficient Map

C-scan maps useful for determining elastic property variations, large defects, homogeneity of entire sample

Ultrasonic Transducers

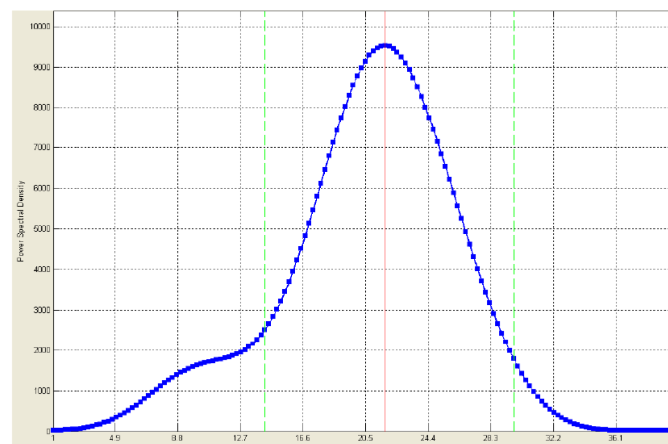


<u>Piezoelectric Effect</u>:

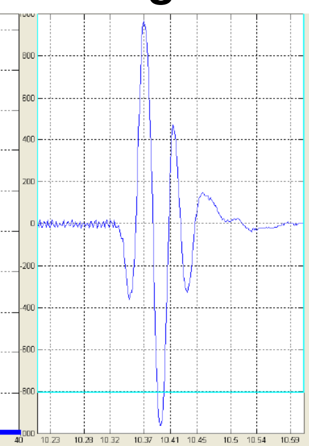
Electric Potential ⟺ **Crystal Deformation and Oscillation**

- Used to emit and receive ultrasonic pulses
- Pulse created by piezoelectric crystal or ceramic
- Each transducer emits over a range of frequencies dependent on piezoelectric crystal size, geometry, and backing material

Transducer Output

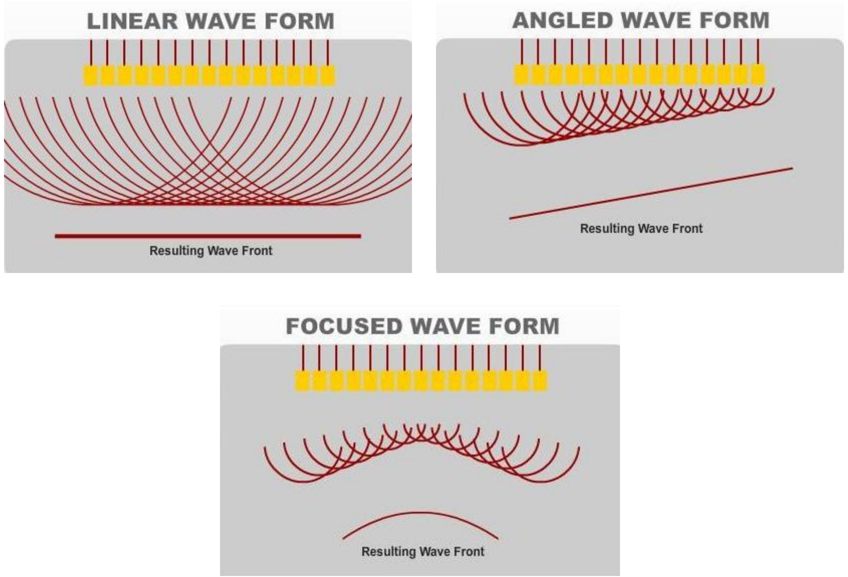


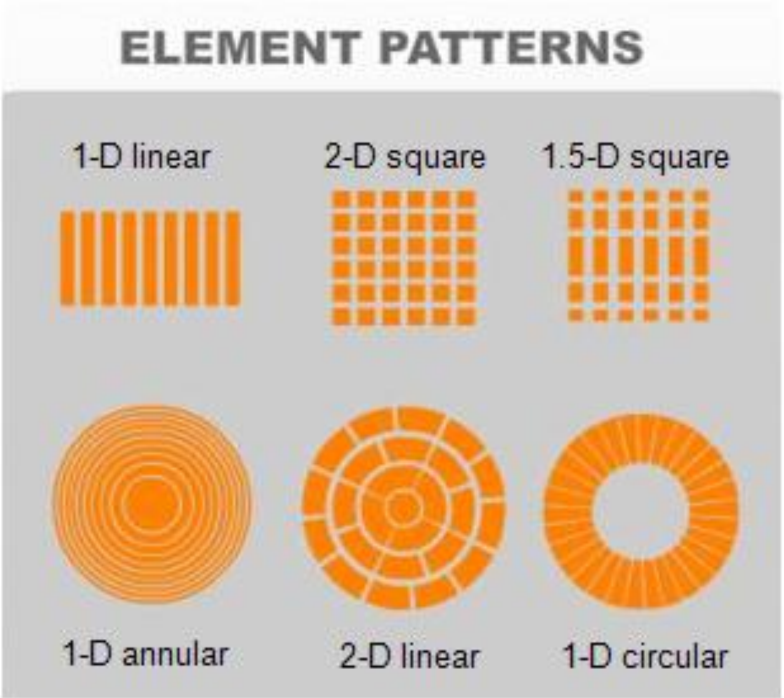
Signal

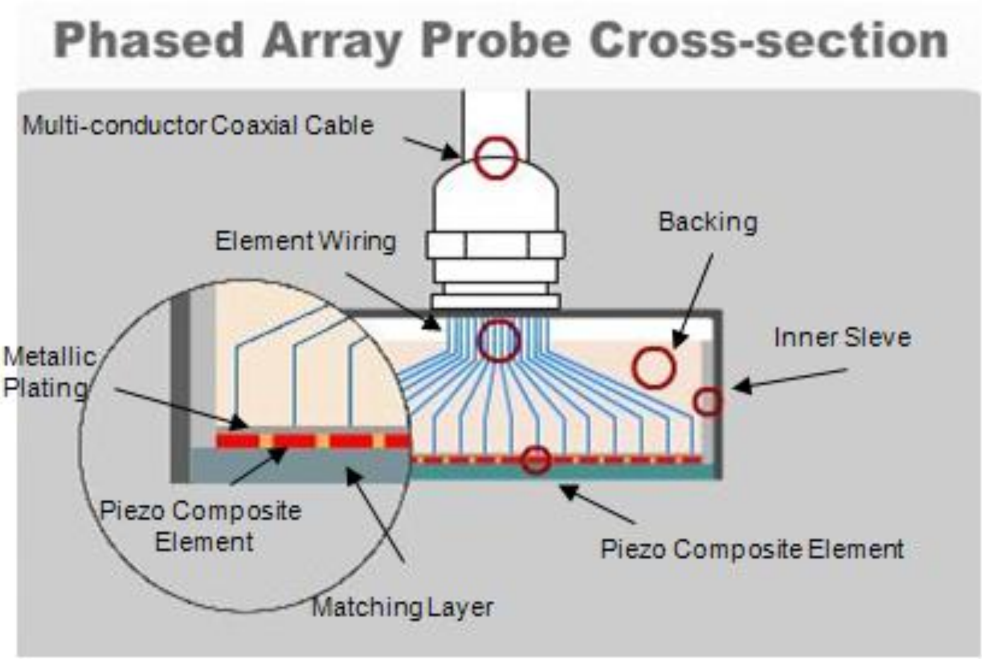


Phased Array Fundamentals

- Probe consists of multiple elements, controlled independently
- Beam profile adjusted by firing elements at different times
- Focus beam on points or planes of different depths
- 10x faster than conventional ultrasonics





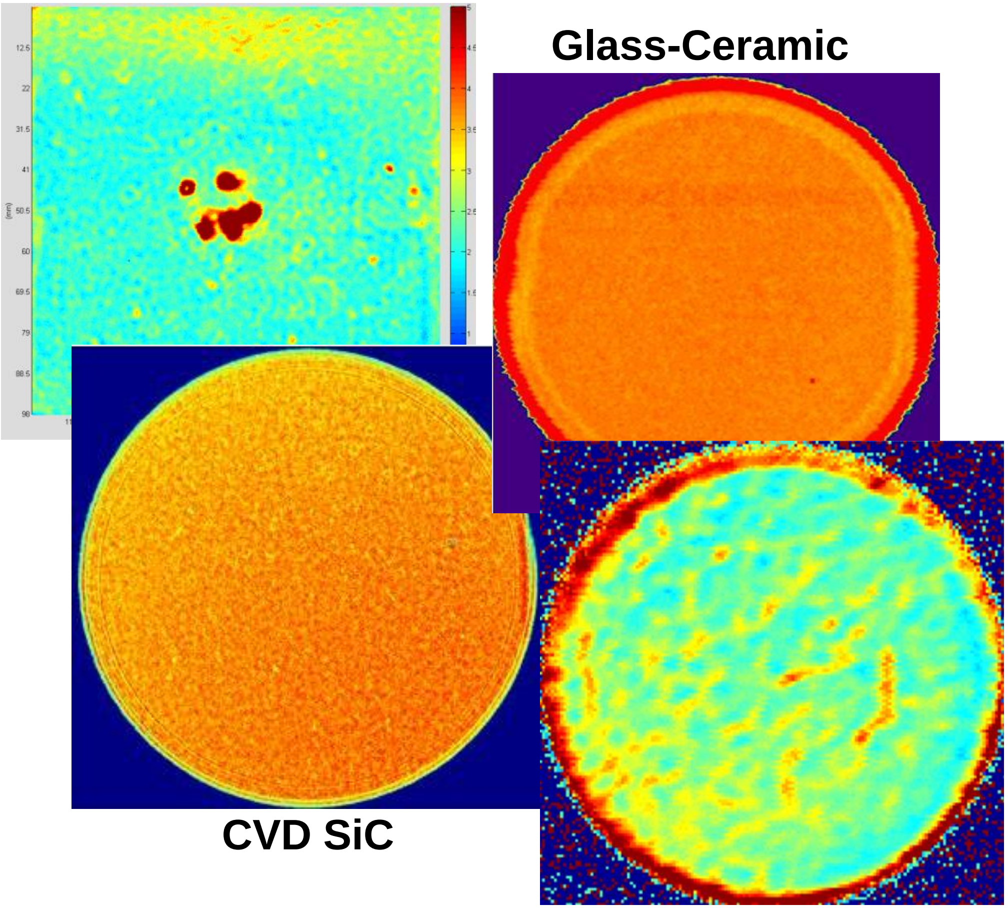


*Images taken from Olympus-ims.com

Where NDE Take Us and Where It Stops

Sintered SiC

Glass-Ceramic



CVD SiC

Reaction Bonded SiC

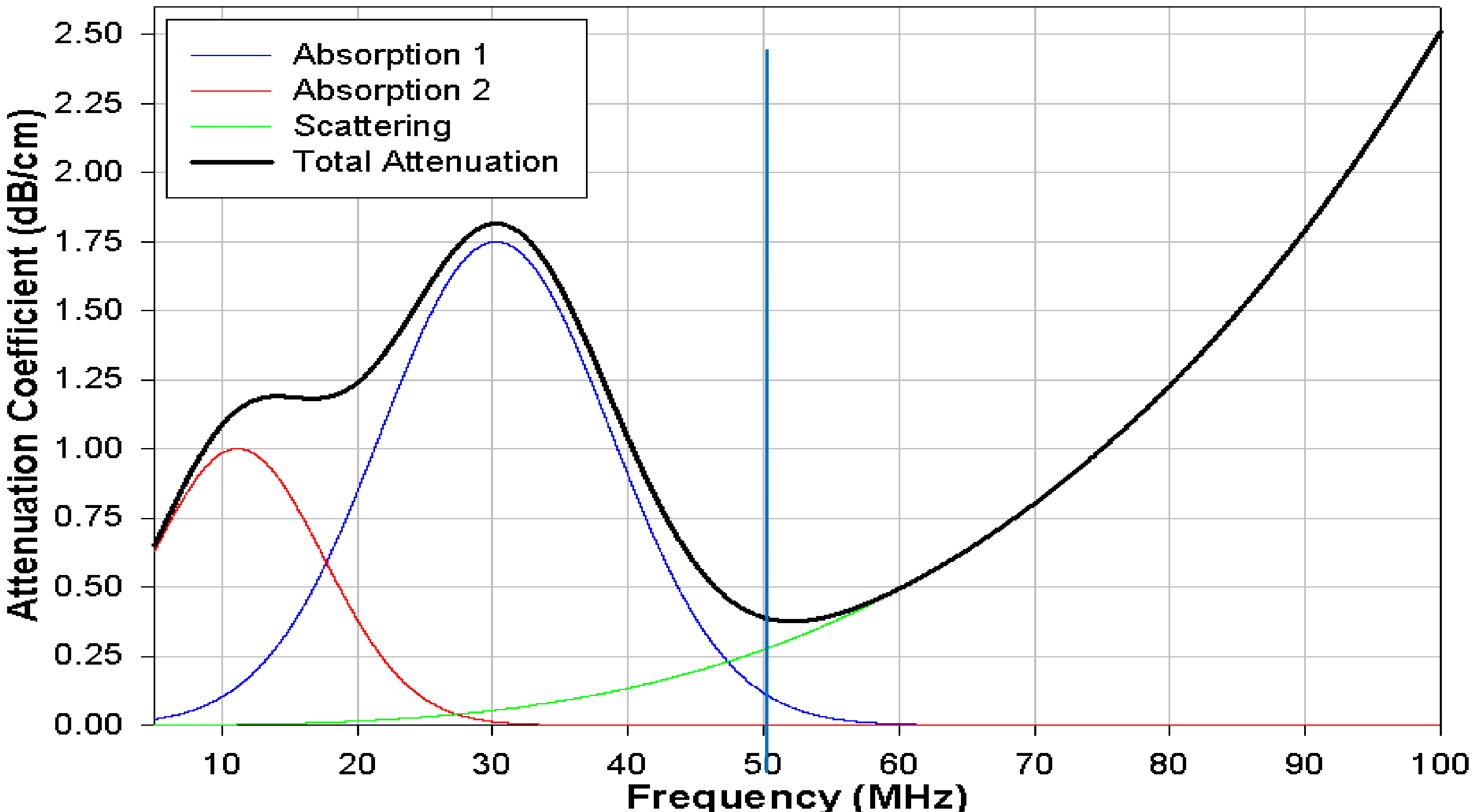
- C-Scan mapping of acoustic velocity and amplitude variations
- Rapid identification of anomalous defects

NDE tells us *where* and *how* sample properties vary

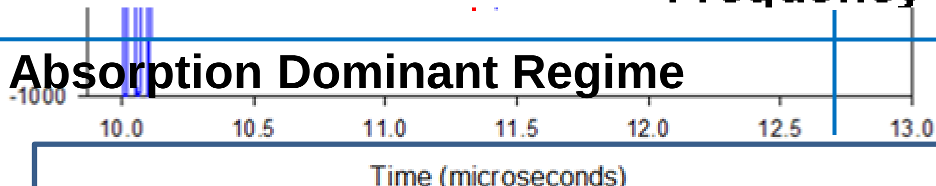
- NDE identifies anomalous defects
  - Composition?
  - Effect on local microstructure?
- NDE measures elastic properties
  - Relate to density
  - New batch compositions introduce elements that reduce density but improve microstructure
- Which values are ‘good’?
- What is the cause of variations?
- Are features seen in UT detrimental to performance?
- Do subtle variations matter?
- Feature Size?

NDE leaves us with *unanswerable* questions

Characterization-Based Measurements



Absorption Dominant Regime **Scattering Dominant Regime**



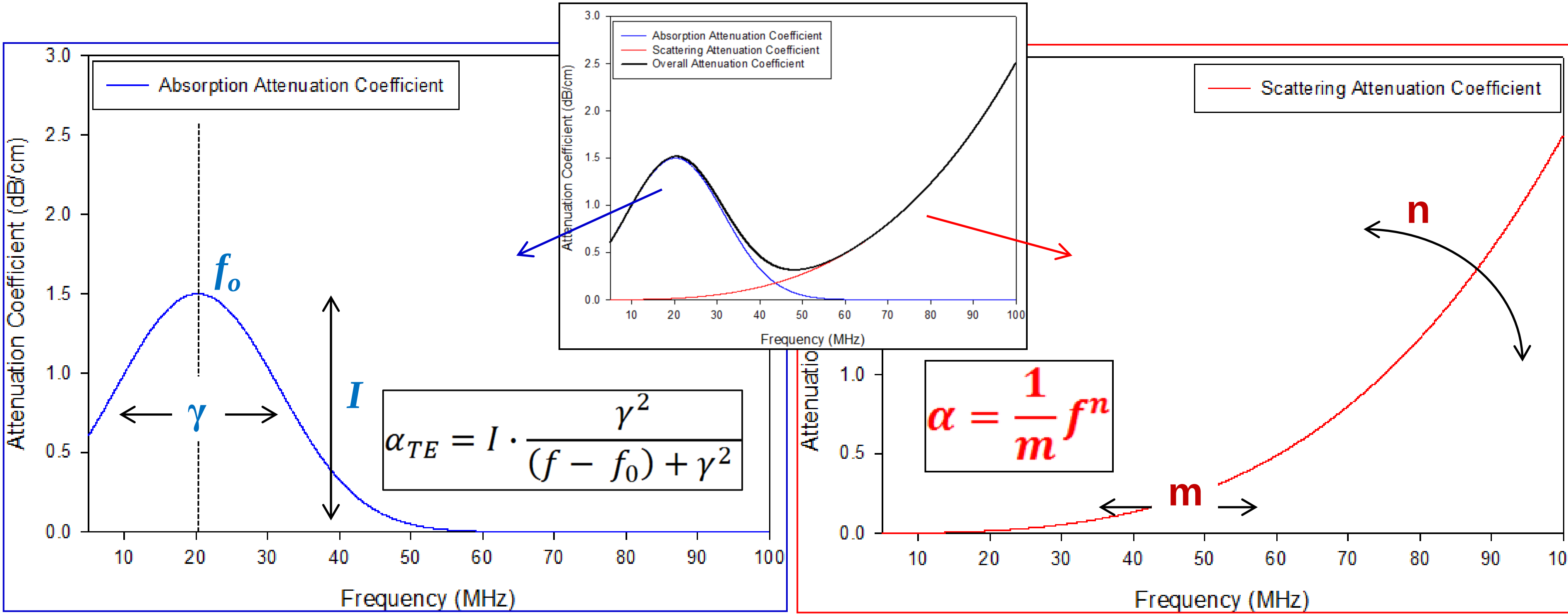
Approximate absorption and scattering regimes for silicon carbide

Absorption: up to ~50MHz when grains are <10µm

Analogous to XRD, etc. – Need Multiple Standards

Scattering: Dominates higher frequencies

Acoustic Attenuation Regimes



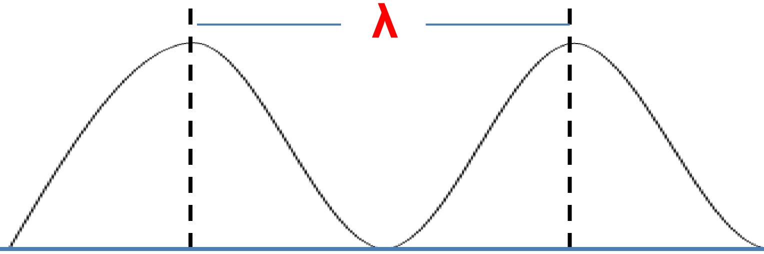
- I → concentration
- f_o → feature type and mean size
- γ → pinning condition

- m → elastic mismatch and morphology
- n → mean size

Absorption at low frequencies → Peak-like behavior

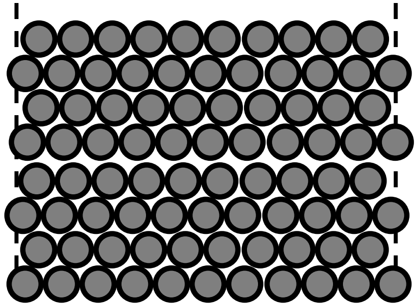
Scattering at high frequencies → Power-law behavior

Wavelength to Scatterer Size ~ Visual Representation



In SiC, wavelength is 150 – 1200μm
for frequencies between 10 – 80MHz

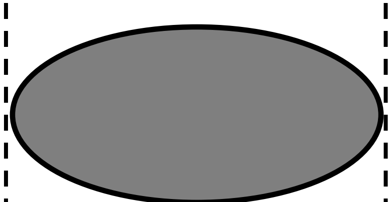
α: attenuation coefficient
a: mean grain size
f: frequency
C parameters: scattering constants



Rayleigh Regime

λ > a
When a is less than approximately λ/10

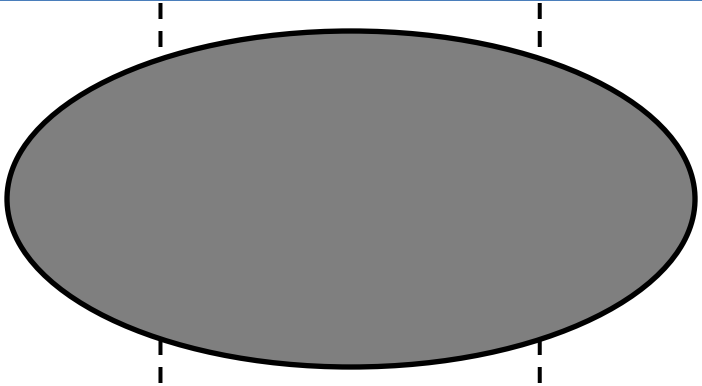
$$\alpha = C_R a^3 f^4$$



Stochastic Regime

λ ≈ a
When a is approximately the same size as λ

$$\alpha = C_S a f^2$$



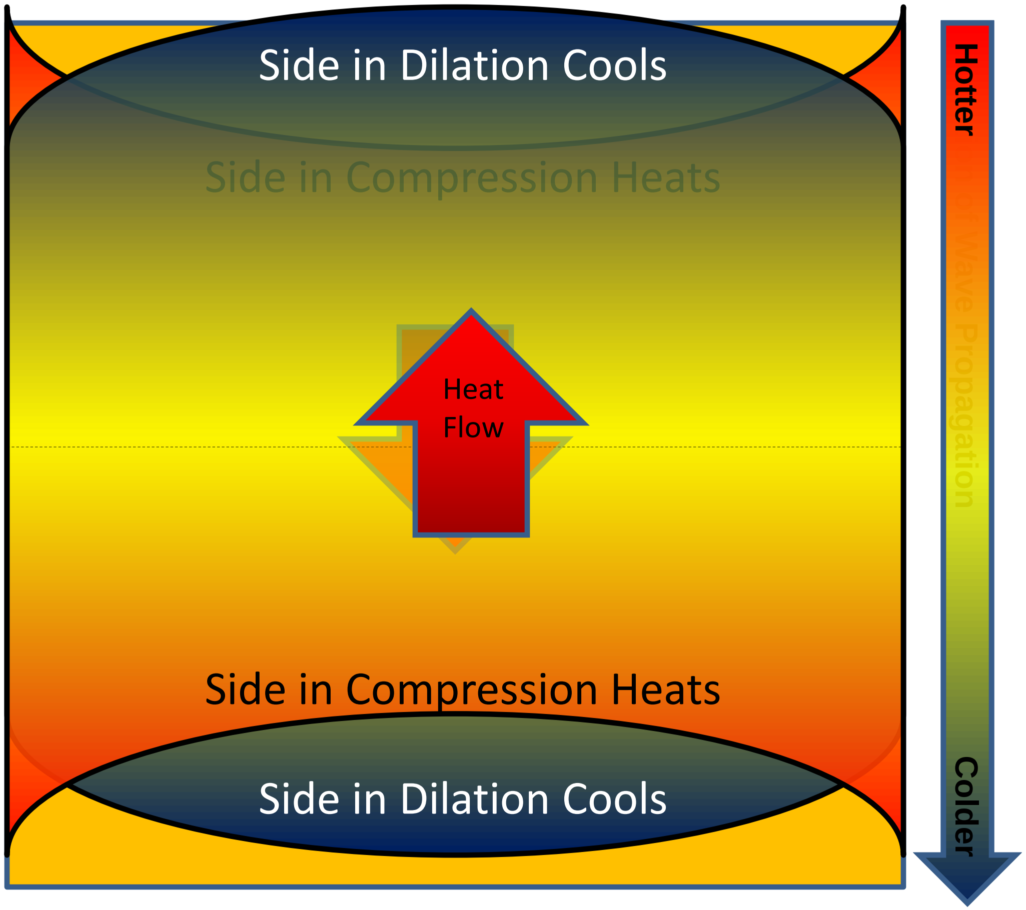
Diffuse Regime

λ < a
When a is larger than λ

$$\alpha = \frac{C_D f^0}{a}$$

[1] Nicoletti, D.W., N. Bilgutay, and B. Onaral, *Scaling Properties Of Attenuation And Grain Size.* Ultrasonics Symposium, 1990 **1051-011**(90): p. 1119-1122

Thermoelastic Intraparticle Absorption



Acoustic wave passes through particle
State of Particle at ¾λ

Particle is bound on both ends elastically by adjacent grains

- As wave propagates, bound particle bends
- One side of the particle in compression, the other side in tension
- Differential heating occurs → thermoelastic effect
- Equilibrium only reached by taking energy from acoustic wave, causing attenuation
- Frequency of maximum attenuation defined as:

$$f_o = \frac{\pi X}{2 \rho C_V a^2}$$

Zener Equation

X – thermal conductivity

ρ – density

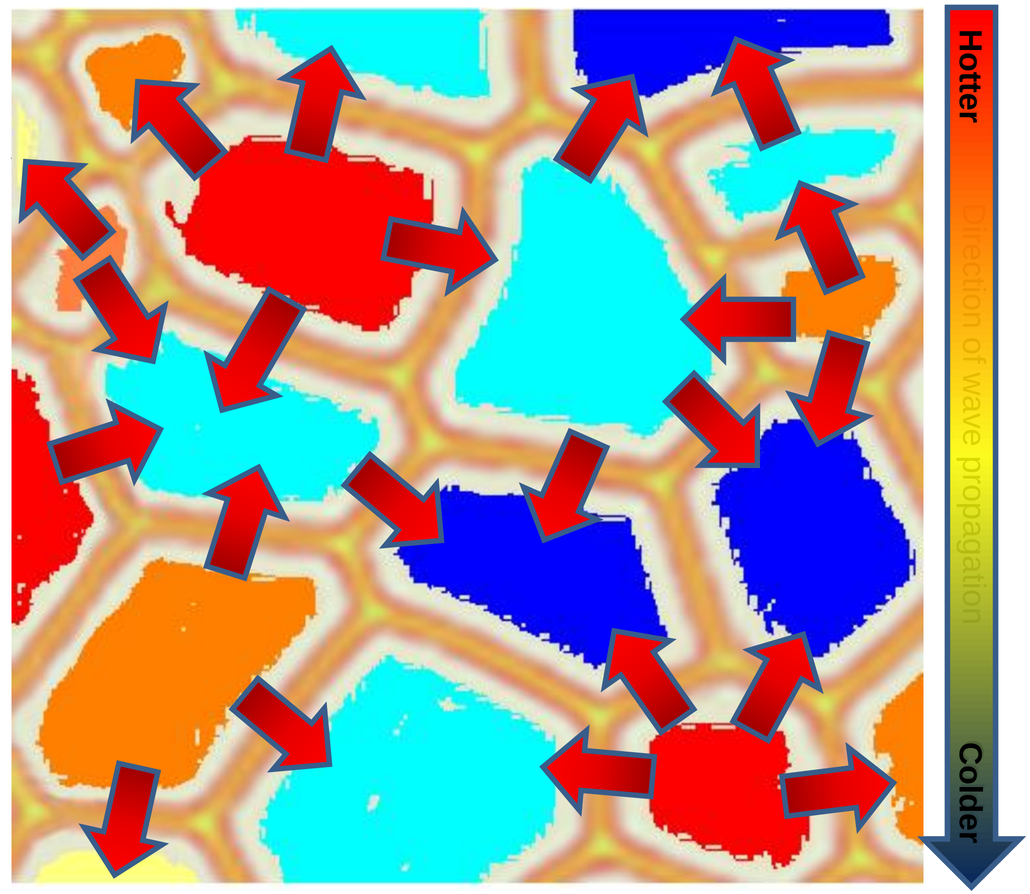
a - diameter

C_V – specific heat

Thermoelastic Interparticle Absorption

- Acoustic wave propagates from a single direction
- Anisotropy in properties leads to differential heating depending on orientation
- Irreversible heat flow occurs
- Energy required for equilibrium taken from acoustic wave, causing attenuation
- Frequency of maximum attenuation defined as:

$$f_o = \frac{3\,\pi\,X}{2\,\rho\,C_V\,a^2}$$



X – thermal conductivity

ρ – density

a - diameter

C_v – specific heat

Which mechanisms are active?

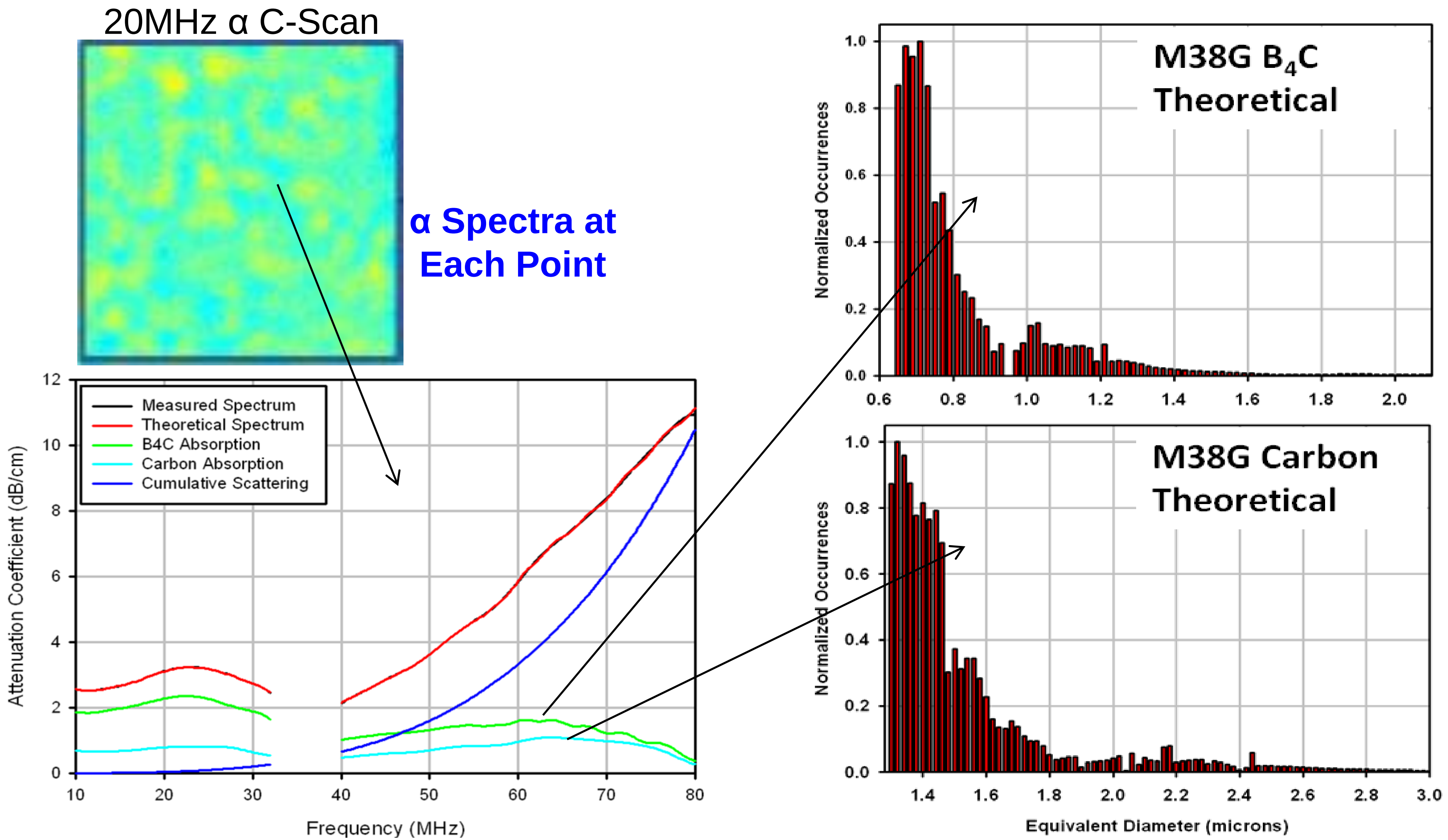
Is this a concern for biphasic materials?

Zener, C., *Internal Friction in Solids.* Proceedings of the Physical Society, 1940. **52**: p. 152-167

Bhatia, *Ultrasonic Absorption*. 1967, Oxford: Clarendon Press

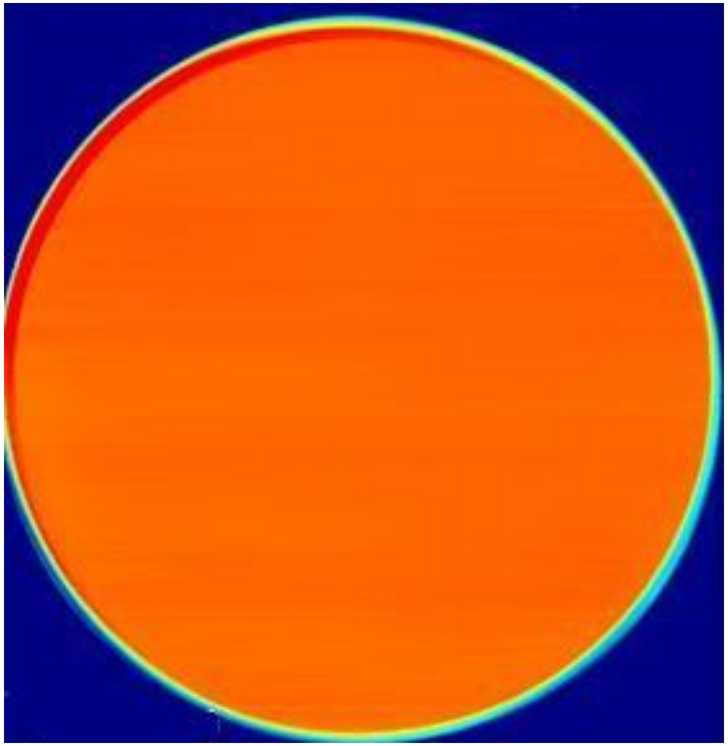
Previous Acoustic Spectroscopy Results

Fully Dense Monolithic Sintered Silicon Carbide

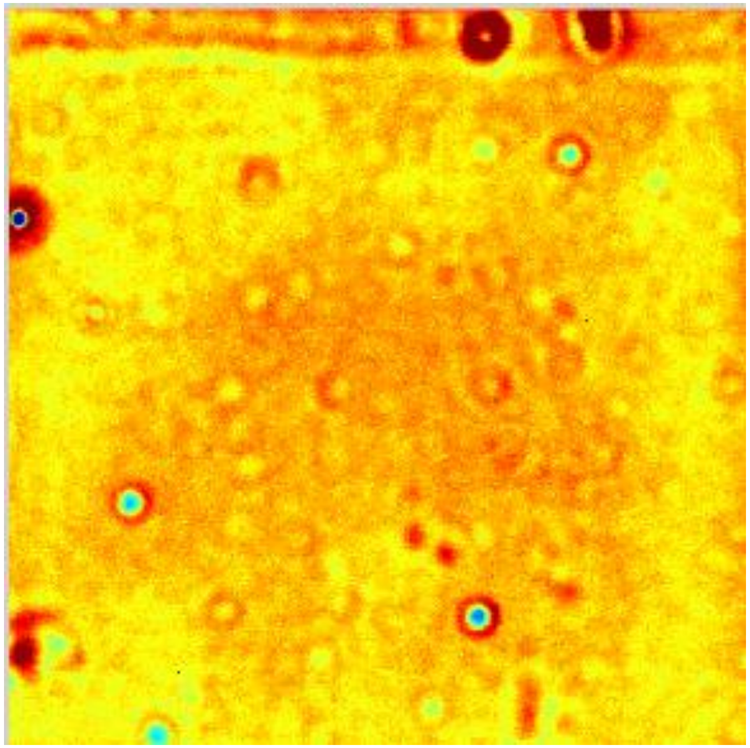


Automated Least Squares Regression Analysis for Predicted Solid Inclusion Size Distributions

Motivation for Nondestructive Testing of Mirror Materials



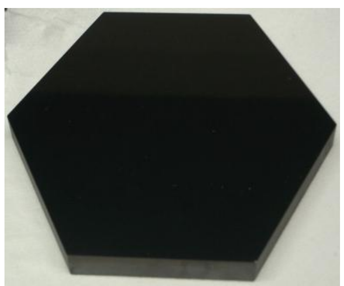
Can we determine quality before deployment?

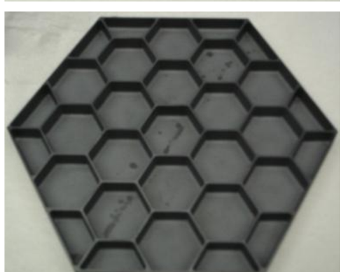


- Cost
  - Evaluate before polishing
    - Go/ no-go criteria for continuing
  - Evaluate after polishing
    - Determine errant polishing defects, degree of polishing
- Failure
  - Location of large anomalous defects
  - Microstructural characterization of grain, solid inclusion size
    - Effects of thermal , mechanical stress, vibratory stresses

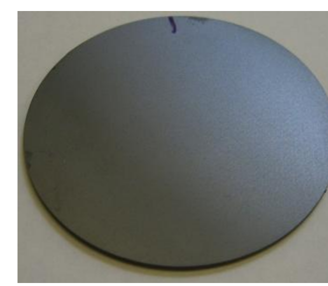
Samples

Sample	Manufacturer	Material	Condition	Form
I, II, III	St. Gobain	Sintered SiC	unfinished	lightweighted, planar blanks
01	SSG	RB-SiC	polished	lightweighted, planar mirror
02	M Cubed	RB-SiC	polished	lightweighted, planar mirror
03	Schafer	SiC foam core, Si coating	polished	lightweighted, planar mirror
A, B, C, D	SSG	RB-SiC	polished	planar flats

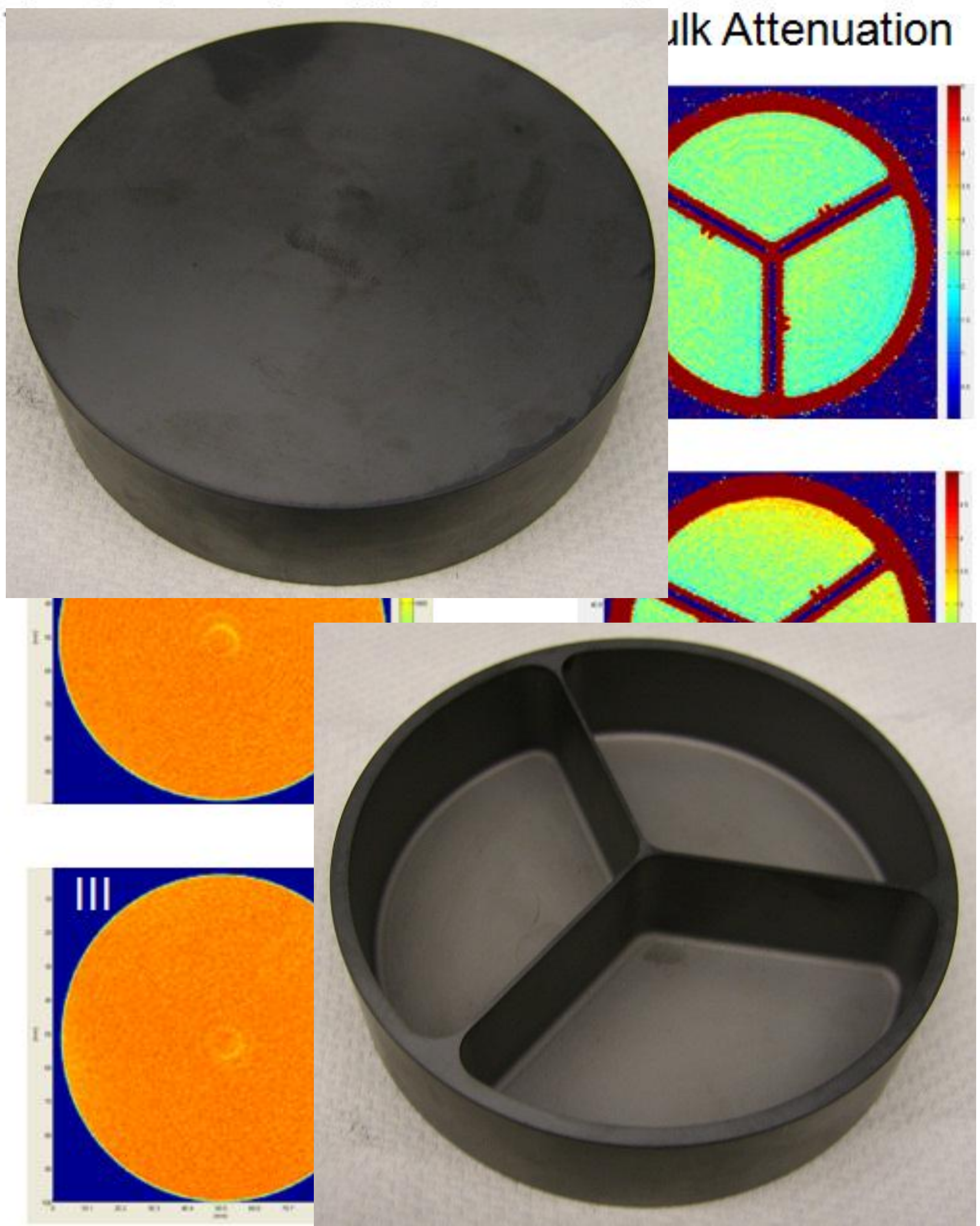




How Can We Evaluate These?

Ultrasound Nondestructive Testing of Mirror Blanks

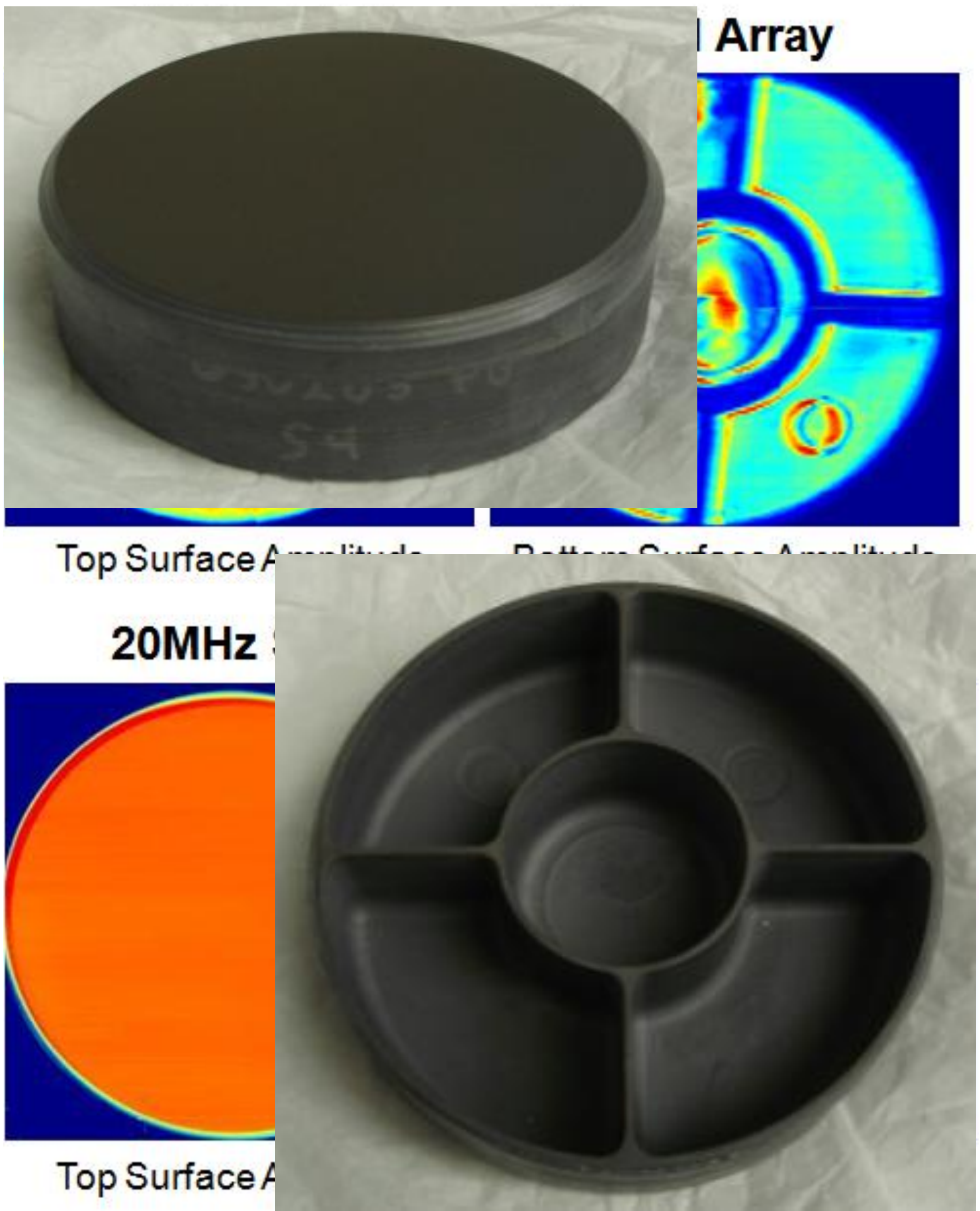
Sintered SiC Lightweighted Unfinished Planar Blanks



- 20MHz C-Scan maps to determine:
  - Surface Irregularities
  - Bulk Defect
- Sintered silicon carbide mirror blanks
  - As ground, before polished
- Show relative uniformity and reproducibility between samples
- Any large irregularities caused by sample support structure
- Relate NDE before and after polishing

Comparison of Single Element and Phased Array

Lightweighted Planar Polished Mirrors



- Phased array C-Scans performed much faster than single element
- Phased array detects more surface/subsurface irregularities
- Both phased array and single element show similar results for bottom surface

---

- Phased array shows scanning artifacts and distortion of images
- Phased array probes limited to relatively low frequencies compared to single element transducers
- Single element transducer shows much higher spatial resolution

Ultrasound Evaluation for Subsurface Uniformity

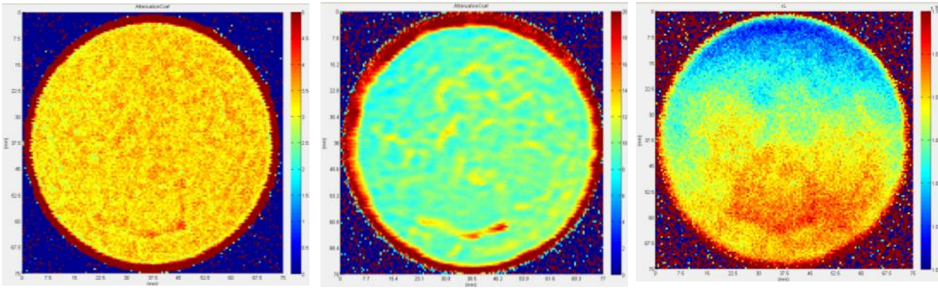
Single Element Amplitude Testing of Various SiC Mirrors

Amplitude gradient → Density, thickness gradient

Location of large defects

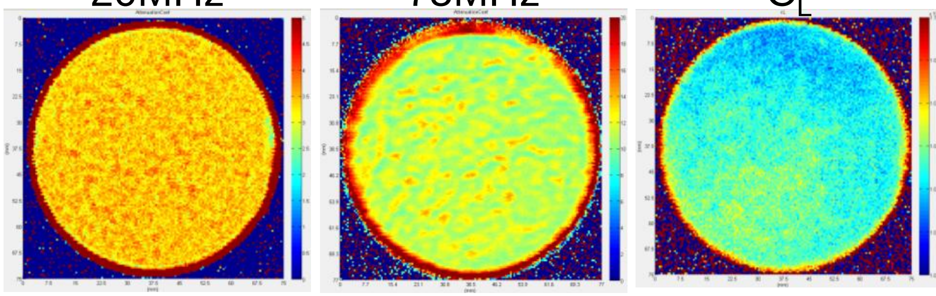
Reaction Bonded SiC Mirror Samples

A



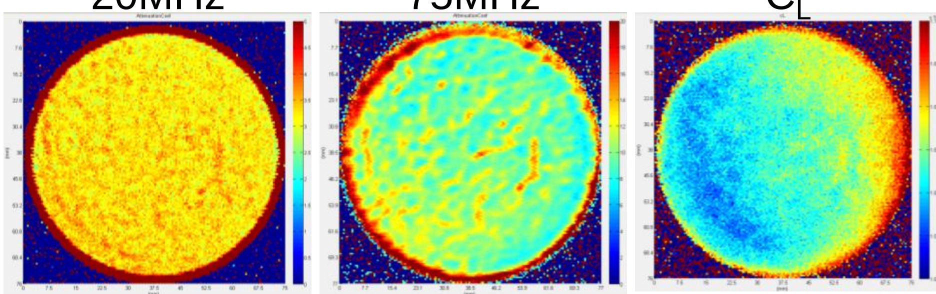
20MHz 75MHz C_L

B



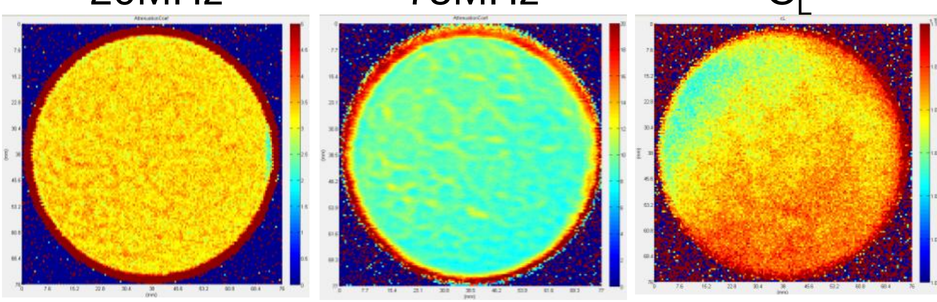
20MHz 75MHz C_L

C



20MHz 75MHz C_L

D

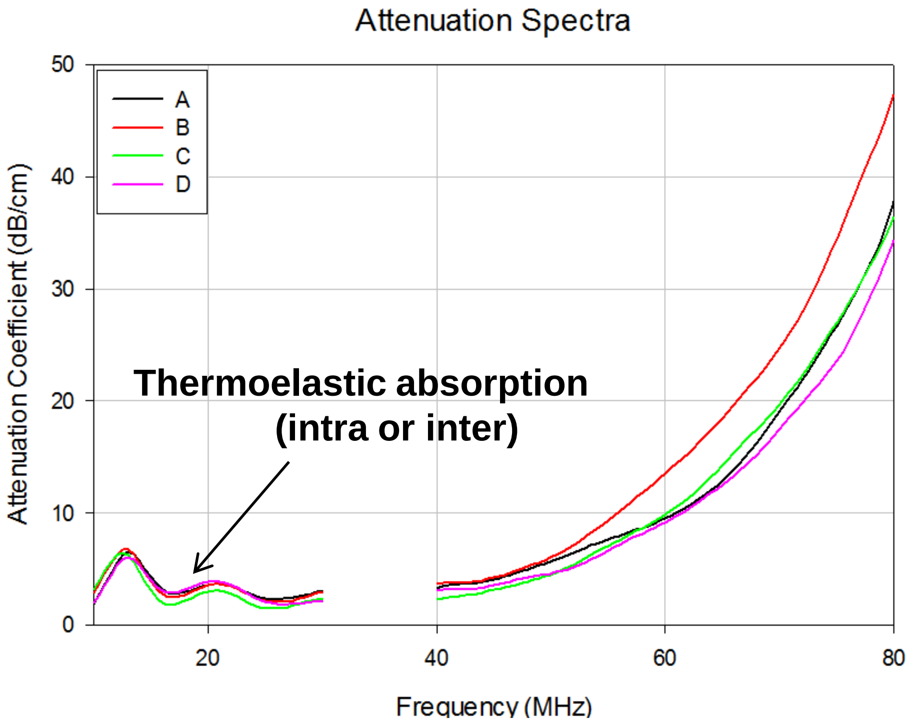


20MHz 75MHz C_L

Reaction Bonded SiC Polished Planar Flats

- **Attenuation coefficient show variability in bulk material**
  - At 20MHz, appear as darker red regions of higher attenuation within lighter orange and yellow background
  - Clearer at 75MHz, features of yellow or orange in lighter blue/green background
- **Inverse relationship with α and C_L**
  - Indicates presence of unreacted silicon within microstructure → lower speed of sound, high α in those regions
  - Gradient in speed of sound → possible gradient in density

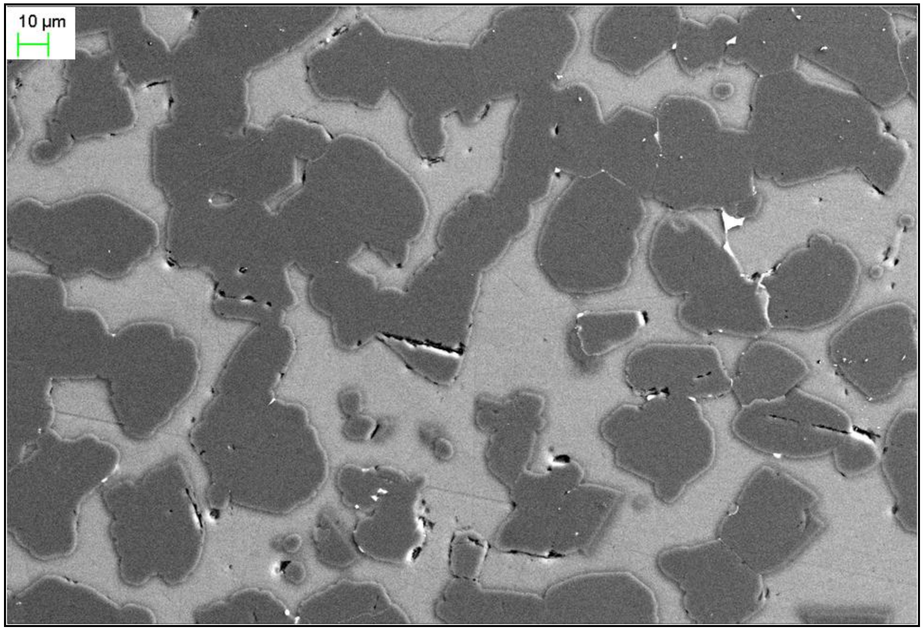
Reaction Bonded SiC Mirror Samples



Absorption between 11 – 23MHz

- Silicon grains:
  - Intraparticle: 2.5 – 3.6µm
  - Interparticle: 7.5 – 10.8µm
- SiC grains:
  - Intraparticle: 2.2 – 3.1µm
  - Interparticle: 6.6 – 9.3µm

- Power law exponent of ~4 - indicative of predominantly Rayleigh scattering
- Suggests grain size is much smaller than the wavelength (135 – 270µm), so the SiC grains should 5 – 10x smaller: 13.5 – 54µm
- **Attenuation appears to be caused by interparticle absorption by Si grains and scattering by SiC grains**



Summary

- Reviewed features that can be observed with high resolution using ultrasound:
  - Surface and subsurface irregularities using amplitude scans
  - Bulk defects using attenuation coefficient scans
- Ultrasound can be used to nondestructively characterize mirror materials prior to polishing and deployment
  - Microstructural information can be used to predict if the mirror will perform well in service

State of the Art

- Single transducers with frequencies >100MHz, using attenuation measurements to determine grain size, composition
  - Limited to flat surfaces, regular geometry
- Phased array can find acoustically different phases using conventional ultrasound methods, even on curved surfaces
  - Currently limited to lower frequencies

Acknowledgements

Ceramics, Composite, and Optical Materials Center

ARL Materials Center of Excellence – Lightweight Materials for Vehicle Protection

USAF Grant No. FA8650-08-C-5205

St. Gobain, SSG, M Cubed, Schafer